

1986

ANNUAL QUALITY ASSURANCE PERFORMANCE REPORT

SECTION 6

SOIL LEACH SAMPLES

INORGANIC TRACE CONTAMINANTS SECTION

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G. C. RONAN, DIRECTOR
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Ministry of the Environment

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1986

ANNUAL QUALITY ASSURANCE PERFORMANCE REPORT

SECTION 6

SOIL LEACH SAMPLES

INORGANIC TRACE CONTAMINANTS SECTION

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Inorganic Trace Contaminants Section
Laboratory Services Branch
Ministry of the Environment

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INORGANIC TRACE CONTAMINANTS SECTION

SUMMARY

I. Introduction

The Inorganic Trace Contaminants Section of the Ministry of the Environment, Laboratory Services Branch is responsible for the analysis of a wide variety of sample types for metals and non-metals. The use of sensitive instrumentation and methodologies appropriate to the sample matrix, combined with quality assurance programs, ensures that the Section is able to maintain a high standard of analytical performance. This performance is monitored through regular internal quality control and assurance programs as well as participation in interlaboratory round-robins. This QA report summarizes the methodologies used for analysis of these samples and the supporting internal quality assurance data.

This report is assembled in sections that reflect the analyses performed on different sample matrices in support of the programs of the Ministry of the Environment. Coincidentally, these divisions also reflect the supervisory responsibilities within the Section.

II. Quality Control and Assurance

The objectives of the quality control and assurance programs are to ensure that all of the components of the analytical process are under control and to ensure immediate detection and correction of unacceptable analytical performance. The program monitors all of the reagents, instrumentation, calibration and recovery components of the analytical system.

A. Quality Control

Quality control of the analytical process takes place at the instrument level and is intended to ensure that the instrumentation is operating according to established criteria. This control function ensures that instrument calibration, standardization, slope and intercept, and instrumental drift meet these criteria.

B. Quality Assurance

Quality assurance of the analytical process takes place after the results have been generated and is intended to ensure that the analytical protocols of sample preparation and digestion have been carried out correctly. This control function ensures that reagent blanks, digested standards, sample duplicates and recovery materials meet established response criteria.

III. Report Format

The report consists of one page method summaries and one page data summaries of blanks, between-run controls and within-run duplicates in formats that are common to all of the parameter/matrix combinations. The method summaries give a brief outline of the sample preparation and measurement procedures. The data summaries consist of annual mean values with standard deviations.

For the within-run duplicates, the data set is subdivided into ranges approximating 0 to 20 %, 20 to 50 % and 50 to 100% of the analytical range. All results for duplicates reported to the data base that are "<" or that have been diluted into the range are excluded from the statistical analysis.

The standard deviations for blanks and between-run controls are calculated using formula I. Formula II is used for the calculations for within-run duplicates.

$$sd = \sqrt{[(\text{sum}x^2 - (\text{sum}x)^2)/n/(n-1)]} \dots\dots I$$

$$sd = \sqrt{(\text{sum}d^2/2n)} \dots\dots II$$

where : x = the individual values; n = the number of events
d = the differences between pairs of duplicates

The data is stored in a personal computer using BMB Manager II files. All data manipulations, reports generated etc, are performed using applications written in Manager Math.

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6. Soil Leachate

6.1 Soil leachates are samples created in the lab as a result of leaching solid samples with water or other simulated natural aqueous solvent mixtures. The purpose of this sample pretreatment is to determine the potential of metals and other parameters to be leached from the material under environmental conditions.

Table 6.1 summarizes the parameters determined, the preparation methods used and the instrument types used for the analysis of soil leachates.

TABLE 6.1

Parameter	Collection Device	Preparation	Analysis
Metals	Plastic jars	Acid leach	ICP - AES
Anions	Plastic jars	Water ext	IC
Mercury	Plastic jars	Acid leach	Cold vapour AAS
Hydride Metals	Plastic jars	Acid leach	Hydride AAS

6.2 Soil Leachate Quality Assurance

Sub aliquots of soil samples may be leached separately to generate duplicate leach samples. Blanks consist of the leaching solution used on a sample.

TABLE 6.2

Sample Designation	Type	Parameter
chk 5	Synthetic matrix matched spiked sample	ICP Metals
chk3	Synthetic matrix matched spiked sample	ICP Metals
comp7	composite leachate (limited use)	ICP Metals

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: ALUMINUM TEST CODE: ALUT SAMPLE TYPE: Soil Leachate

UNIT: Spectroscopy/Forensic SUPERVISOR: D. Boomer

METHOD CODE: 001AE5

REVISION NO: 85-1

DATE: December 30, 1985

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 100 ml

Container- Polyethylene bottle with screw top

Preservative- 1% HNO₃

Other- Samples are leached in the field and then sent to the lab.

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-
Procedure- Samples are analyzed straight without pretreatment by the

Elan ICP-MS Spectrometer.

INTERFERENCES: Calcium at high concentrations

REPORTING RESULTS: 4 decimal places

INSTRUMENTATION: Elan 250 ICP-MS

Calibration Range: 0.0001 to 0.500 mg/L

Resolution: 1 a.m.u.

Sensitivity:

Instrument Detection Limit: 0.00007 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.0001 to .500 mg/L

Accuracy-

Precision of Controls-

	A	B
mean	.0263mg/L	.0255
std. dev.	.0012mg/L	.0014
R.S.D.	4.6 %	5.4 %

Precision of Duplicates-	low range	mid range	high range
s.d.	0.0071	0.0382	0.0146
mean	0.0617	0.1626	0.3617

W .002 mg/L

T .020 mg/L

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

ALUMINUM IN SOIL LEACHATE

Operating Range = .00010 to 0.500 mg/L

IN - RUN DUPLICATES

Range	<.00010	.00010 to 0.100	0.100 to 0.250	0.250 to 0.50	> 0.50
no.	1	3	8	11	2
s.w.		0.00710	0.03820	0.01460	
mean		0.06170	0.16260	0.36170	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
CHK5	3	0.02630	0.00120	4.56
chk3	22	0.02550	0.00140	5.49
COMP7	0	0.00000	0.00000	0.00

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	.00000	.00000

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: COPPER TEST CODE: CUUT SAMPLE TYPE: Soil Leachate

UNIT: Spectroscopy/Forensic SUPERVISOR: D. Boomer

METHOD CODE: 001AE5

REVISION NO: 85-1

DATE: December 30, 1985

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 100 ml

Container- Polyethylene bottle with screw top

Preservative- 1% HNO₃

Other- Samples are leached in the field and then sent to the lab.

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-
Procedure- Samples are analyzed straight without pretreatment by the
Elan ICP-MS Spectrometer.

INTERFERENCES: Calcium at high concentrations

REPORTING RESULTS: 4 decimal places

INSTRUMENTATION: Elan 250 ICP-MS

Calibration Range: 0.0001 to 0.100 mg/L

Resolution: 1 a.m.u.

Sensitivity:

Instrument Detection Limit: 0.00004 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.0001 to .100 mg/L

Accuracy-

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

%

Precision of Duplicates-low range

mid range

high range

s.d. 0.0004

0.0007

mean 0.0027

0.0215

W

T

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

COPPER

IN SOIL LEACHATE

Operating Range = .00010 to 0.100 mg/L

IN - RUN DUPLICATES

Range	<.00010	.00010 to 0.020	0.020 to 0.050	0.050 to 0.10	> 0.10
no.	13	6	1	0	0
s.w.		0.00040	0.00070	0.00000	
mean		0.00270	0.02150	0.00000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
CHK5	0	0.00000	0.00000	0.00
chk3	0	0.00000	0.00000	0.00
COMP7	0	0.00000	0.00000	0.00

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	.00000	.00000

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: IRON TEST CODE: FEUT SAMPLE TYPE: Soil Leachate

UNIT: Spectroscopy/Forensic SUPERVISOR: D. Boomer

METHOD CODE: 001AE5

REVISION NO: 85-1

DATE: Dec 30, 1985

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 100 ml

Container- Polyethylene bottle with screw top

Preservative- 1% HNO₃

Other- Samples are leached in the field and then sent to the lab.

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-

Procedure- Samples are analyzed straight without pretreatment by the

Elan ICP-MS Spectrometer.

INTERFERENCES: Calcium at high concentrations

REPORTING RESULTS: 4 decimal places

INSTRUMENTATION: Elan 250 ICP-MS

Calibration Range: 0.0001 to 0.100 mg/L

Resolution: 1 a.m.u.

Sensitivity:

Instrument Detection Limit: 0.005 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.0001 to .100 mg/L

Accuracy-

Precision of Controls-

	A	B
mean	.0725mg/L	.0749
std. dev.	.0035mg/L	.0075
R.S.D.	4.8 %	9.4 %

Precision of Duplicates-low range mid range high range

s.d.	0.0058	0.0034	0.0090
mean	0.0145	0.0279	0.0807

W .005 mg/L

T .050 mg/L

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

IRON

IN SOIL LEACHATE

Operating Range = .00010to 0.100 mg/L

IN - RUN DUPLICATES

Range	<.00010	.00010to 0.020	0.020to 0.050	0.050to 0.10	> 0.10
no.	12	5	8	6	3
s.w.		0.00580	0.00340	0.00900	
mean		0.01450	0.02790	0.08070	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
CHK5	26	0.07250	0.00350	4.83
chk3	49	0.07490	0.00700	9.35
COMP7	5	0.06580	0.04200	63.83

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	8	.00300	.00250

DATE 87/07/13

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: MANGANESE TEST CODE: MNUT SAMPLE TYPE: Soil Leachate

UNIT: Spectroscopy/Forensic SUPERVISOR: D. Boomer

METHOD CODE: 001AE5

REVISION NO: 85-1

DATE: December 30, 1985

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 100 ml

Container- Polyethylene bottle with screw top

Preservative- 1% HNO₃

Other- Samples are leached in the field and then sent to the lab.

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-
Procedure- Samples are analyzed straight without pretreatment by the

Elan ICP-MS Spectrometer.

INTERFERENCES: Calcium at high concentrations

REPORTING RESULTS: 4 decimal places

INSTRUMENTATION: Elan 250 ICP-MS

Calibration Range: 0.0001 to 0.100 mg/L

Resolution: 1 a.m.u.

Sensitivity:

Instrument Detection Limit: 0.00004 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.0001 to .100 mg/L

Accuracy-

Precision of Controls-

	A	B
mean	.0250mg/L	.0262
std. dev.	.0012mg/L	.0017
R.S.D.	4.8 %	6.4 %

Precision of Duplicates-	low range	mid range	high range
s.d.	0.0004	0.0241	0.0025
mean	0.0620	0.0336	0.0787

W .002 mg/L

T .020 mg/L

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

MANGANESE IN SOIL LEACHATE

Operating Range = .00010 to 0.100 mg/L

IN - RUN DUPLICATES

Range	<.00010	.00010 to 0.020	0.020 to 0.050	0.050 to 0.10	> 0.10
no.	6	19	5	2	2
s.w.		0.00040	0.02410	0.00250	
mean		0.00620	0.03360	0.07870	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
CHK5	26	0.02500	0.00120	4.80
chk3	50	0.02620	0.00170	6.49
COMP7	5	0.10400	0.04820	46.35

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	6	.00010	.00000

ANALYTICAL PROCEDURE

Inorganic Trace Contaminants Section

TEST NAME: NICKEL

TEST CODE: NIUT

SAMPLE TYPE: Soil Leachate

UNIT: Spectroscopy/Forensic

SUPERVISOR: D. Boomer

METHOD CODE: 001AE5

REVISION NO:85-1

DATE: December 30, 1985

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 100 ml

Container- Polyethylene bottle with screw top

Preservative- 1% HNO3

Other- Samples are leached in the field and then sent to the lab.

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-
Procedure- Samples are analyzed straight without pretreatment by the

Elan ICP-MS Spectrometer.

INTERFERENCES: None

REPORTING RESULTS: 4 decimal places

INSTRUMENTATION: Elan 250 ICP-MS

Calibration Range: 0.0001 to 0.100 mg/L

Resolution: 1 a.m.u.

Sensitivity:

Instrument Detection Limit: 0.00007 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.0001 to .100 mg/L

Accuracy-

Precision of Controls-

A

B

mean

std. dev.

R.S.D.

%

Precision of Duplicates-low range

mid range

high range

s.d. 0.0004

```
mean      0.0056
```

W

T

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

NICKEL

IN SOIL LEACHATE

Operating Range = .00010to 0.100 mg/L

IN - RUN DUPLICATES

Range	<.00010	.00010to 0.020	0.020to 0.050	0.050to 0.10	> 0.10
no.	16	4	0	0	0
s.w.		0.00040	0.00000	0.00000	
mean		0.00560	0.00000	0.00000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
CHK5	0	0.00000	0.00000	0.00
chk3	0	0.00000	0.00000	0.00
COMP7	0	0.00000	0.00000	0.00

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	.00000	.00000

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: ZINC TEST CODE: ZNUT SAMPLE TYPE: Soil Leachate

UNIT: Spectroscopy/Forensic SUPERVISOR: D. Boomer

METHOD CODE: 001AE5

REVISION NO: 85-1

DATE: December 30, 1985

NATURE OF LAST REVISION:

SAMPLE HANDLING:

Quantity Required- 100 ml

Container- Polyethylene bottle with screw top

Preservative- 1 % HNO₃

Other- Samples are leached in the field and then sent to the lab.

SAMPLE PREPARATION: Partial Extn.- Total Extn.- % Extracted-

Procedure- Samples are analyzed straight without pretreatment by the
Elan ICP-MS Spectrometer.

INTERFERENCES: Ca at high concentrations

REPORTING RESULTS: 4 decimal places

INSTRUMENTATION: Elan 250 ICP-MS

Calibration Range: 0.0001 to 0.100 mg/L

Resolution: 1 a.m.u.

Sensitivity:

Instrument Detection Limit: 0.00006 mg/L

PERFORMANCE CHARACTERISTICS:

Routine Operating Range- 0.0001 to .100 mg/L

Accuracy-

Precision of Controls-

	A	B
mean	.0305mg/L	.0736
std. dev.	.0010mg/L	.0115
R.S.D.	3.3 %	15.6 %

Precision of Duplicates-low range mid range high range

s.d.	0.0004	0.0063	0.0018
mean	0.0078	0.0279	0.0590

W .002 mg/L

T .020 mg/L

CONTROL LIMITS:

REMARKS:

SUMMARY REPORT OF QUALITY CONTROL DATA

ZINC

IN SOIL LEACHATE

Operating Range = .00010 to 0.100 mg/L

IN - RUN DUPLICATES

Range <.00010 .00010 to 0.020 0.020 to 0.050 0.050 to 0.10 > 0.10

no.	0	6	22	5 SD	1
s.w.		0.00040	0.00630	0.00180	
mean		0.00780	0.02790	0.05900	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
CHK5	26	0.03050	0.00100	3.28
chk3	50	0.03130	0.00250	7.99
COMP7	5	0.07360	0.01150	15.62

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	6	.00020	.00010



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